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STRESS-CORROSION CRACKING OF HIGH-STRENGTH ALLOYS

Contract DA-04-495-ORD-3069

Structural Materials Division

NOV 8 7962



AZUSA, CALIFORNIA

INVESTIGATION OF STRESS-CORROSION CRACKING OF HIGH STRENGTH ALLOYS

Contract DA-04-495-ORD-3069

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CONTRACT FULFILIMENT STATEMENT

This is the seventh in a series of quarterly progress reports submitted in partial fulfillment of the contract.

It constitutes the first quarterly progress report for the one-year continuation of the original two-year program.

I. OBJECTIVES

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The objectives of the program extension are outlined below:

- A. To investigate the stress corrosion cracking characteristics of at least three new high strength alloys of interest for rocket motor case applications. These alloys will be 6Al-4V titanium, 18-Nickel Maraging steel, and 20-Nickel Maraging steel, in addition to limited testing of vacuum-melted 9Ni-4Co steel.
- B. To study the environmental parameters that could affect the rate and extent of stress-corrosion cracking.
- C. To determine the effect of material parameters composition, strength level, welding and microstructure on stress-corrosion susceptibility.
- D. To continue the evaluation of protective coatings and other techniques for preventing stress-corrosion cracking.

II. SUMMARY

The program for the third-year effort has been outlined. In addition to the bent-beam and U-bend test methods, an accelerated test method employing notched tensile specimens will be introduced. Procurement orders for all test materials have been placed and one alloy shipment has been received. This alloy, 6A1-4V titanium, is under evaluation for chemical composition and mechanical property requirements.

III. WORK PROGRESS

A. ALLOYS TO BE TESTED

Since the initiation of the original test program two years ago, a number of new high strength alloys have been receiving increased attention in the

aircraft and missile field due to their extremely favorable mechanical properties. The effort for the third year will be directed to the study of three steel alloys and one titanium alloy.

1. 6A1-4V Titanium, AMS 4911

This high strength titanium alloy is presently being widely used for pressure vessels and rocket motor cases. It has excellent fabricability and mechanical properties. We propose to test this alloy under three metallurgical conditions. These conditions will be: annealed; quenched and aged to maximum strength; and welded. The heat treatment to attain maximum strength will most likely be a 1650°F solution anneal, water quench, and 900°F age. Our initial heat treatment on flat tensile specimens resulted in some distortion. Straightening by re-flattening of the samples which had been quenched and aged would greatly add to the machining costs, as well as contributing undesirable residual stresses. A special fixture is now being fabricated that will avoid this problem. The quenched specimens will be clamped flat into the fixture and aged. The residua... stresses will be relieved by creep-relaxation giving us flat stress-free samples. Tensile, bent-beam, U-bend and center-notch specimens have been rough-machined so that heat-treatment, final machining, and environmental testing will begin soon. Chemical and mechanical property tests of as-received sheet have been conducted and it was found that the material conforms to Specification AMS4911, as shown in Table 1. Specimens of titanium 6A1-4V alloy will be subjected to a post-heat treat machining operation to remove the oxide layer which forms during heat treatment. It has been found that removal of at least 0.010 in. from each surface of the material is required after heat treatment to restore full ductility to this alloy.

2. 20-Nickel Maraging Steel

This is a high strength steel that will attain desired strengths with a single aging treatment. Sufficient material is on order for testing under the following conditions:

a. Solution annealed, refrigerated at -100°F for 16 hours; aged at 850°F for 4 hours; air cooled

- b. 50% Cold-worked; aged 850°F for 1 hour; air cooled
- c. 75% Cold-worked; aged 850°F for 1 hour; air cooled
- d. Welded; solution annealed; refrigerated to -100°F for 16 hours; aged at 850°F for 4 hours; air cooled

(Expected strength levels are shown in Column 2 of Table 2.)

3. 18-Nickel Co Mo Maraging Steel

This steel is similar to the 20-nickel maraging steel noted above. In addition to the high-strength and notch-toughness properties it is reported by the International Nickel Company to have exceptional resistance to stress-corrosion-cracking. Sufficient material for complete testing, at the following four strength levels is on order:

- a. Solution annealed aged 900°F for 3 hours and air cooled. Titanium content 0.3 to 0.6%
- b. 50% Cold-worked; aged 900°F for 3 hours; air cooled. Titanium content 0.3 to 0.6%
- c. 50% Cold-worked; aged 900°F for 3 hours; air cooled. Titanium content 0.6 to 1.0%.
 - d. Welded; solution annealed; aged 900°F for 3 hours; air cooled. (Expected strength levels are indicated in column 2 of Table 2.)

4. <u>9Ni-4Co Steel</u>

This is a vacuum-melted steel; it is not yet being used commercially. This material is reported to have exceptional notch toughness and high yield and ultimate strengths. The material will be tested at 2 or 3 strength levels. Heat treatments necessary to attain these strength levels will be furnished by the supplier.

B. COATINGS EVALUATION

Protective coatings will be tested as they are received from vendors. Specimens will be coated by commercially developed processes which have shown

promise for corrosion protection of metals exposed to the environments under study in this program. To obtain a valid coating evaluation, the base metal must be highly susceptible to stress corrosion cracking. Consequently, H-ll steel, which showed such susceptibility in earlier tests, will be used. Specimens will be stressed after coating application so that the evaluation of coating will include its ability to be flexed.

C. TEST METHODS

1. Bent-Beam Test

The bent-beam test will continue to be the primary test method used in the program. In order to prevent any galvanic effects, between the specimen and the holder, the specimen will now be insulated from the holder by supporting the ends with polycarbonate blocks, attached to the stainless steel bar. Polycarbonate resin has excellent resistance to all the water solution environments and will allow no stress relaxation of the specimen. Figure 1 shows the Phelps-type holder after modification to insulate the specimen.

2. <u>U-Bend Test</u>

The U-bend test, shown in Figure 2, will be used to a more limited extent. The U-bend will be employed to accelerate failure times in the tests where the center notch test cannot be used (i.e., for high-humidity and outdoor environments). It will also be used in the trichloroethylene environment.

3. Center-Notch Test

The test specimen configuration to be used for accelerated testing is shown in Figure 3. It consists of a 1-3/4- by 8-in. tensile specimen containing a central notch. This notch is made by a two-step process: first, Elox-machining of a 0.06- by 0.57-in. slot extended at each end by very narrow Elox-machined notches 0.001-in. root radii, and second, extension of these notches by fatigue cycling to produce fatigue cracks of controlled dimensions.

Such specimens have been used at Aerojet in stress-corrosion evaluations with excellent consistency of results and short failure times. Table 3

illustrates some failure times obtained with AMS 6434 material tempered at various temperatures and exposed to a tap water environment. No failure occurred when this alloy was exposed to air under identical conditions.

Stressing of the specimens is conducted in a creep machine; the test environment is applied by filling a polyethylene cup cemented to the specimen.

4. Test Environments

The test environments are shown in the master plan, Table 2. The only new environment added to the extension program is the seacoast atmosphere exposure. This exposure was suggested by Mr. H. Rosenthal of Frankfort Arsenal on his Aerojet visit of October. These tests will most likely be conducted at the Aerojet-General Oceanics facility at Newport Beach, California. All bent-beam and U-bend tests will be conducted, in triplicate, for a minimum of 1000 hours. The center-notch tests will be conducted, in duplicate, for a minimum of 100 hours.

IV. FUTURE WORK

It is expected that the 20-Ni and the 18-Ni Maraging steels will be received within the next 3 weeks, while the last alloy to be evaluated, the 9Ni-4Co steel will be received within the next 8 weeks. Specimens for all phases of the titanium 6Al-4V evaluation have been machined and heat treating is now in progress. Some stress corrosion test data for all of the alloys to be tested will be available by the next quarterly report period. Test fixtures have been prepared with polycarbonate blocks attached to the ends of the Type 347 stainless steel bars to eliminate any galvanic effects between the specimen and the holder as shown in Figure 1.

TABLE 1

CHEMICAL AND MECHANICAL PROPERTIES OF 6A1-4V TITANIUM

Chemical Analysis

	C	_A1_	<u>v</u>	05	N ₂		Ti	<u>Fe</u>	Other
Mill Report	.03	6.1	4.1	-	.014	.80 ppm	Rem	.16	.18
AGC Check	-	6.3	4.6	.083	.015	60 ppm	Rem	.26	-
AMS 4911 Max.	.10	6.75	4.5	.15*	.05	150 ppm	-	.30	.40
Min.	_	5.50	3.5	-	-	•	-	-	-

Mechanical Properties (Annealed Material, Transverse)

	Yield Strength (0.2%-Offset)	Tensile Strength	Elongation	Hardness
Mill Report	131,900	141,400	12%	Re 33.5
AGC Test	138,000	143,800	14%	Re 34
AMS 4911	120,000 min.	130,000 min.	10% min.	Re 36 max

^{*}Accepted standard for oxygen, but not part of specification.

TABLE 2

TENTATIVE MASTER PLAN FOR THIRD ANNUAL PROGRAM

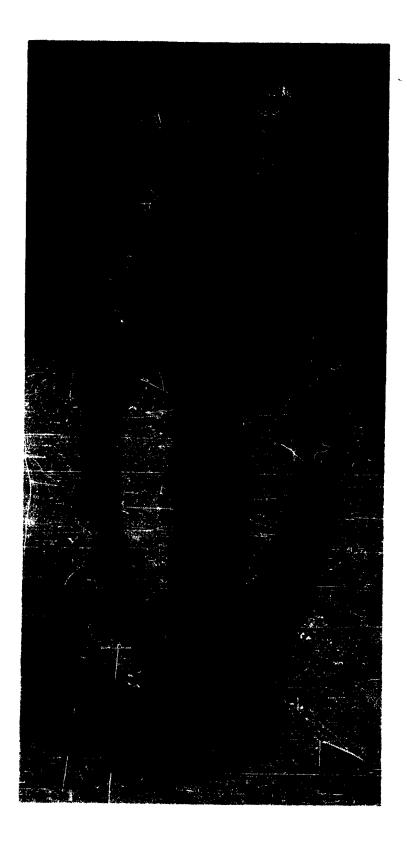
				•	Distilled			Sodius Dichromate	Soluble	4	140				,
Alloy	Acticipated 0.2% Yield Strength	Possible Beat Trestment	Pettod Metbod	Code	-01)	- (S)	2 2 2 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3	Solution (-04)	(-9 (-5)	b	etavlene	Compline (-08)	Propellant (-09)	14 Atr	Property (-11)
GA1-kV Titanium	135, 000	As received, annealed	Ment Bear U-Bend Center Entob	G-1-B G-1-U	‡ _~	n i	- n:	nic	n.,			~.	~.	•	_
	170,000	1650°F 40 and 900°F age	Bent Mess U-Mend Center Motch	6-2-0 6-2-0 6-2-0	וחימ		א ורחומ	w m i a	w m 1 a		ı ını	1 M 1 :	i Mi	N 10 10	i mm
	110,000	Helded, 900°P age	Bent Beam U-Bend	6-Y-5 6-Y-0	· • ·	m i	. m :	ı mı	1 m 1	. WW	n	. IO. 1	I M I	ייייני	ı mm
20-Heisel Maraging Steel	250,000	Solution angeal -100°F, 850°F age	Bent Beam U-Bend Center Notch	H-1-B H-1-U H-1-C	w i a	n.	m 1 8	K I W	m i a	in in i	in i	n.	K) I	n.,	n mm
	275,000	50\$ CH 850% age	Bent Bear U-Bend Center Motch	H-2-B H-2-U H-2-C	r i a	* \ 1 \ 1	m 1 0	n i w	i n i a	nn 1	~ 1	m, i	· M ·	v m i c	2
	300,000	75% C# 850°F age	Nent Beam U-Mend Center Rotch	#-3-B #-3-0 #-3-0	nia	m : 1	IN F Q	יייומי	וראומ	WW I	M) i	ווחו	' n i		\^~
	250,000	No 1 ded	Nent Beam U-Bend	H-V-B	ı, n	m i	ואו	ı m ı	ı n ı	m.,	~		, "	~ ~	n i m
18-Rinco Managing Steel	340,000	05 CM 0.3/0.6 ≸ TH Aged at 900 F	Nent Beam U-Nend Center Notch	I-1-B I-1-U I-1-C	m i a	w ·	rn i a	mia	m : a	K) K)	110	n i	\	• •	PO BAM
	300,000		Bent Beas U-Bend Center Notch	1-2-B 1-2-U	m + 0	W) 1 1	. 	ו ויאו ו			110	n n i	n	1 W PA	5 I 68
	80°08	50% CW 0.6/1.0% Th Aged at 900°P	Bent Beam U-Bend Center Motch	1-3-B 1-3-U 1-3-C	יתימ	m 1 1	m i a	· ~ 1 &	וחימ	n.	110		, , m,	10 5	's #
	No data on hand		Bent Beam U-Bend	I-N-B I-N-U	~ ·	٠,	₩.1	ı Ka ı	.	m m				1 (N) 11"	3 "
981-ACo M.cuan-Chat	No data on band	Aged 0.25%/0.30% C	Bent Bear U-Bend Conter Botch	J-1-E	מומ	۳.	n i (m i i	m + (₩.,	. In	المراز أ	NI M	\	. "
And the second				1.2.1	•		±. √ 4	ф. ,	4	1		7	•	. . •	-

TABLE 3

STRESS-CORROSION CRACKING OF CENTER NOTCH SPECIMENS

(Typical previous Aerojet data with AMS 6434 steel exposed to tap water. All samples austenitized 1-1/2 hour at 1650°F, quenched in 375° salt and tempered 2 hours at the indicated temperatures.)

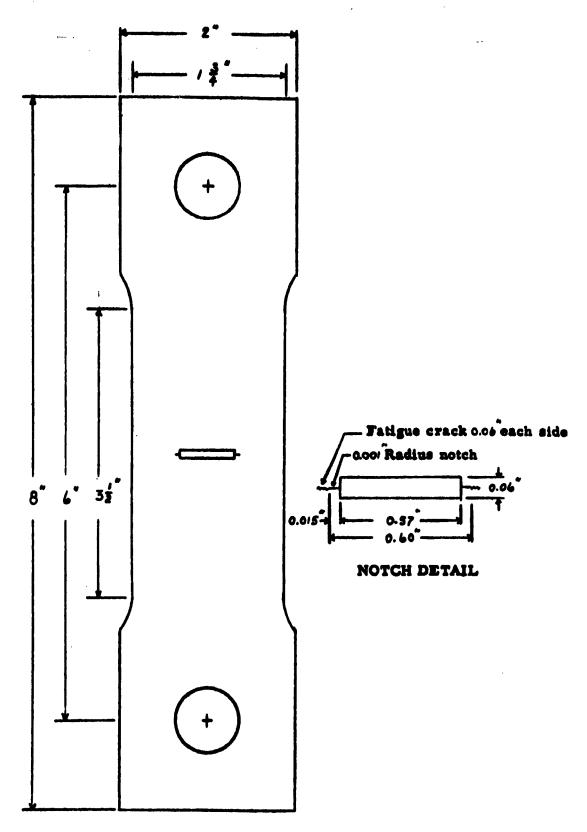
Sample No.	Tempering Temperature	Stress in % of Notch Tensile Strength	Failure Time, hours
11A	550° F	88.1	1.3
14A	***	87.3	1.3
15A	tt	88.7	0.6
11A	600°F	88.5	0.8
12A	11	88.7	1.4
15A	11	90.4	1.9
11A	650 ⁰ F	87.2	5.9
13A	11	91.0	4.2
15A	11	88.9	2.7
10A	700°F	89.5	8.1
11A	II .	88.7	8.0
13A	n .	88.7	7.1
9A. '	800°F	92.4	19.9
10A	11	90.0	16.2
17A	11	89.1	31.8



Insulated Phelps Bent-Beam Specimens



Figure 2



ELOX-NOTCHED SPECIMEN FOR CRACK PROPAGATION STUDY

Figure 3